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DEVELOPMENT OF NOVEL DECONTAMINATION TECHNIQUES  
FOR EXPLOSIVE CONTAMINATED FACILITIES  
Contract DAAK11-81-C-0101

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1.0 INTRODUCTION

One of the United States Army Toxic and Hazardous Materials Agency's (USATHAMA) mandates is to restore contaminated Army facilities which have been used for manufacturing, loading, packing and storing explosives so that these facilities can be removed or reclaimed for alternate use. Explosive contaminants of concern include TNT, 2,4-DNT, 2,6-DNT, HMX, RDX, Tetryl and other explosives manufactured and used by the Army. Facilities to be addressed include the building production equipment, above and underground storage tanks, wastewater/sludge sumps ventilation ducts, conduits and related explosive/munition production. The decontamination involves the removal of explosives from exposed surfaces of the materials as well as explosives that have penetrated porous media, cracks, and expansion joints. Site inspections have been performed and the contaminated structures include a wide range of concrete and wood frame structures.

1.1 The 5X Dilemma

The desired level of decontamination to be achieved is "5X" which is defined as "permitting unrestricted use of the previously contaminated site or material". The 5X level of decontamination is also defined as being

exposed to a temperature of 1000 F for 15 minutes. Since the 5X condition has an operational rather than an analytical definition, it does not provide a means to evaluate the relative efficacy of novel decontamination concepts. The only method currently being used to decontaminate building facilities is to torch (or flame) structural components in order to burn the explosive materials. However, no experimental documentation exists that indicates that flaming actually accomplishes total decontamination, although our study indicates that this conclusion is warranted in the case of surface decontamination.

### 1.2 Program Outline

The decontamination of buildings by flaming is only partially effective and is a very expensive technique. Thus, the objective of this program was to define and evaluate a novel decontamination method which would achieve the 5X criteria.

To implement this objective the program was divided into three phases:

- Phase 1. Identify, rank order, and select novel decontamination and inerting concepts for laboratory evaluations (Completed)
- Phase 2. Experimentally validate selected concepts and recommend a concept(s) for field study (In Progress).
- Phase 3. Implement concept(s) on pilot-scale decontamination process in the field. (Future)

### 2.0 PHASE 1 STUDIES

The first activity performed was the identification of potential decontamination and inerting methods. This was performed by performing the following tasks:

- Literature reviews
- Idea generation sessions

- Compilation of a computerized data base cataloging, storage and retrieval system.

These tasks resulted in the generation of potential decontamination concepts which are listed in Table 1 in the general categories of thermal, extractive, abrasive, chemical and inerting methods.

All concepts were evaluated by applying the following evaluation criteria: safety, damage to building, penetration depth, applicability to complex surfaces, operating costs, capital costs, and waste treatment/recovery costs. Further criteria which were utilized to chemical decontamination concepts were mass transfer and destruction efficiency ratings.

Those concepts which were selected for evaluation in Phase 2 studies are shown in Table 2.

### 3.0 PHASE 2 STUDIES

#### 3.1 Enhanced Aqueous Solubilization of Explosives

Each of the three chemical concepts that were tested in these studies are compatible with and typically performed in aqueous based solvent systems. However, each of the six target explosives have very low solubility (about 0.01 percent or lower) in water. Those additives which were screened for their potential enhancement of the aqueous solubilities of explosives included the following materials:

- Cosolvents: dimethylsulfoxide (DMSO), dimethylformamide (DMF), and acetone
- Surfactants: nonionic, cationic, anionic, and speciality surfactants
- Complexing Agents: diethanolamine and 4-hydroxyethylpiperazine

The aqueous cosolvents DMSO and DMF at concentrations of 30 percent or higher were judged most suitable for use as decontamination or extraction solvent systems.

TABLE 1. CONCEPTS EVALUATED IN PHASE I

Thermal	Abrasive
Hot Gases	Vacu-Blast
Radiant (Infrared) Heating	Hydroblasting
Flaming	Acid Etch/Neutralization
Microwaves	Sandblasting
Burning to Ground	Scarification
Flashblasting	Demolition
Electrical Resistance Contact Heating	Cryogenics
Solvent Soak/Controlled Burning	Ultrasonic Extraction
CO <sub>2</sub> Laser	Electropolishing
Hot Plasma	Drill and Spall
Extraction	Chemical
Steam Cleaning - External	Radical Initiated Decomposition
Surfactants	Base Initiated Decomposition
Strippable Coating	Reduction with Sodium Borohydride
Vapor Phase Solvent Extraction	Reductive Cleavage
Steam Cleaning - Manual	Microbial Degradation
Solvent Circulation (FREON)	DS2 Decomposition
Supercritical Fluids	Gamma Radiation
	Sulfur Based Reduction
	Reactive Amines
	Ultraviolet Light/Hydrogen Peroxide
	Molten Reactant Systems
	Active Metals and Acids
	Nucleophilic Displacement
	Ozone Oxidation
	Ascorbate Reduction
	Solid State Hydrogen
Inerting	
	Inerting by Solubilization
	Inerting by Denitration
	Formation of Water Soluble Derivatives
	Desensitization by Water
	Desensitization by Steaming
	Desensitization with Reductants
	Desensitization with Stabilizer Coatings
	Decomposition with Reactive Amines

TABLE 2. SELECTED DECONTAMINATION CONCEPTS

<u>Decontamination</u>		
<u>Thermal</u>	<u>Extraction</u>	<u>Chemical</u>
Hot Gases	Vapor Condensation Solvent Extraction	Radical Initiated Base Initiated Reduction (Sulfur based)
<u>Explosives Inerting</u>		
	Solubilization Water Treatment	

### 3.2 Prescreening of Chemical Decontamination Concepts

The decontamination effectiveness of solutions of sodium hydroxide, sodium sulfide, sodium disulfide, and Fenton's Reagent (catalyzed oxidation) towards explosives in aqueous DMSO and DMF solutions were determined. The variables studied with 2,4-DNT, TNT and RDX were water to solvent ratio, temperature and reagent concentrations. Preliminary product identification studies were performed by using gas chromatography coupled with mass spectroscopy (GC/MS). Sodium sulfide was the most effective decontaminant for all three explosives but was rejected because of the toxic nature of the reaction products (2,4-diamino-6-nitrotoluene from TNT; 2,4-diaminotoluene and nitrohydroxylaminotoluene from 2,4-DNT; and N-nitro-N',N"-bi-(hydroxylamino)hexahydrotriazine from RDX).

The most acceptable chemical decontamination concept for explosives was judged to be solutions of sodium hydroxide in aqueous DMSO (30 or 75 percent DMSO). Preliminary product analysis by GC/MS studies indicated that the major type of products from the decontamination of TNT and 2,4-DNT with sodium hydroxide resulted from the displacement of nitro groups by hydroxide or DMSO anions, the oxidation of methyl groups to carboxylate groups and the dimerization (and subsequent oxidation) to dibenzyl and stilbene derivatives. The major products of RDX and sodium hydroxide are volatile gases and inorganic ions but there was evidence of minor yields of RDX structural isomers.

It should be stressed that structural assignments based on GC/MS analysis are speculative in nature and do not confirm the structure of a compound. Many GC peaks were not identified. Any polymeric material (which has been reported from the reaction between TNT and sodium hydroxide) or other non-volatile products would not be detected by the GC/MS method. Furthermore, under the GC/MS conditions employed, some products may undergo thermal decomposition. Therefore, further analytical efforts are required to confirm the nature of these decontamination products.

### 3.3 Stainless Steel and Concrete Decontamination Results

#### 3.3.1 Analytical Recoveries

Test results indicate that consistently high recovery of explosives can be obtained from stainless steel. However the recovery from concrete is inconsistent and dependent upon the initial explosives concentrations. This interference may be due to a combination of mass transfer and complexation effects as well as the induction of chemical reactions. To confirm adequate removal of explosives from concrete surfaces, a more detailed study of this phenomenon is required.

#### 3.3.2 Hot Gases

Tests have indicated that elevating the temperature of stainless steel, mild steel, and concrete surfaces (both painted and unpainted) to 500 F and maintaining that temperature for 1 hour will remove greater than 99.9 percent of any of the six explosives from all six surfaces which were contaminated. This removal occurs through a combination of volatilization and decomposition pathways.

#### 3.3.3 Chemical Concepts

The results of the solution chemistry studies have been successfully translated to building materials. Decontamination of stainless steel and concrete coupons has been effected by spraying them with 0.1N sodium hydroxide aqueous DMSO solutions.

### 3.4 FUTURE PHASE 2 EFFORTS

#### 3.4.1 Combination of Chemical And Thermal Methods

Pretreatment of explosives spiked coupons with sodium hydroxide solutions (in aqueous DMSO) will be studied to determine if thermal decontamination of explosives will be aided by combining these two approaches.

### 3.4.2 Engineering Analysis

The results of the Phase I site survey and the experimental sub-tasks of Phase II will be incorporated into an engineering/economic analysis of the most promising candidate concepts. This analysis will be the basis for the final recommendations of concepts to be pursued in Phase 3.

## 4.0 PHASE 3

In Phase 3, the concept(s) selected from Phase 2 will be implemented in pilot scale tests at a field site. Contaminated rooms will be sampled, decontaminated and sampled again to determine the effectiveness of the concept. Key concerns in determining the effectiveness of the concept include:

- Sampling methodology
- Analytical methodology
- Validate verification of decontamination
- Level of contamination following treatment (i.e., below detectable limit vs. 5X)
- Data base requirements for full-scale implementation.

## 5.0 CONCLUSIONS

Novel decontamination techniques for facilities are being developed through laboratory and engineering evaluations. A concept(s) will be selected for pilot-scale testing at a field site.